PB-215 083

ANNUAL REVIEW OF LITERATURE (1952) ON ANALYTICAL METHODS PERTAINING TO SEWAGE AND INDUSTRIAL WASTES ANALYSIS

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Cincinnati, Ohio

February 1953

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February 1953

Annual Review of Literature (1952)

on

Analytical Methods

Pertaining to Sewage and Industrial Wastes Analysis

by

Wm. Allan Moore

NATIONAL TECHNICAL
INFORMATION SERVICE

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*An annual review prepared for the
Research Committee
of the
Federation of Sewage and Industrial Wastes Association

Biochemical Oxygen Demand

Busch and Sawyer[10] made a detailed comparison of B.O.D. values obtained when the dissolved oxygen was determined by both the regular Winkler and a polarographic method. In this study, both a synthetic sewage and six types of industrial wastes were used. In the polarographic method a single potential technique was used which eliminated residual current measurements. Only one calibration curve was necessary for the series of different substrates. Waste sulfite liquor was found to interfere with the polarographic dissolved oxygen determination, while both tannery waste and sulfite waste liquor gave low results with the Winkler method. The "k" and "L" values computed from daily determinations on the synthetic media indicated the validity of using the dropping mercury electrode for such studies.

Ingols[28] studied the toxicity of both Cr^{+3} and Cr^{+6} on the B.O.D. as determined by three different methods: (a) dilution B.O.D., (b) manometric B.O.D., and (c) nitrate B.O.D. by nitrate reduction using nitrate as a source of oxygen. Concentrations of Cr^{+3} and Cr^{+6} as high as 20 p.p.m. had little effect on the manometric B.O.D. In the dilution B.O.D., Cr^{+3} was found to be more toxic, whereas with the nitrate method the Cr^{+6} had the greatest effect.

Snow[56] made use of sulfite to remove excess chlorine from chlorinated sewage samples in determining the effect of chlorination on B.O.D. removal. He found that incubated dilutions showed no measurable lag phase due to chlorine toxicity to the seeding organisms and also no measurable oxygen demand due to the excess sulfite. Larionova[35], in studying the effect of trinitrotoluene on biological oxidation, found that 25-80 p.p.m. reduced such oxidation 5-30 percent.

Oxygen Consumed

Czensny[13] made a comparison between the permanganate oxygen consumed and the biochemical oxygen demand, using pure organic compounds. As would be expected in some cases, the oxygen consumed values were higher than the corresponding B.O.D. The author gives a detailed discussion of both parameters and an interpretation of the results obtained by each. Cardone and Compton[11] studied the dichromate oxidation of a number of organic compounds in 50 percent by volume of sulfuric acid. The oxidation was carried out in a boiling water bath, the heating period varying with the type of organic compound being oxidized. The residual dichromate was determined spectrophotometrically at 340 and 850 millimicrons. Theoretical oxidation of some glycols, formaldehyde and formic acid were obtained. Dayres[14], in his study of the permanganate oxygen consumed test, pointed out the importance of keeping the various factors involved constant if concordant results are to be obtained. In the oxidation of ethyl alcohol with dichromate, Ferrari[18] found that the ratio of H_2SO_4/H_2O in the oxidizing mixture must be 1/3 or higher.

Dissolved Oxygen

Freier[19], in the determination of dissolved oxygen in water, substituted o-tolidine for the starch indicator in using essentially the Winkler procedure. Wickert and Ipach[59] reacted dissolved oxygen with ferrous iron in an acid solution in the presence of potassium thiocyanate. The color development was compared with standards containing known amounts of iron.

Kjeldahl Nitrogen

A number of papers were published in the past year on the analytical determination of the various forms of nitrogen. Middleton and Stuckey[46] were able to recover quantitatively as NH_3, the nitrogen from a wide range of heterocyclic compounds by using a Hg catalyst. In their work, they kept the final ratio of Na_2SO_4 to H_2SO_4 constant, by the addition of acid corresponding to the amount of acid consumed in the reaction. Formulae are given for calculating the necessary amount of acid to add. They also added glucose to accelerate the breakdown of simple pyridine derivatives. McCutchan and Roth[41], by using a preheating step with thiosalicylic acid were able to reduce the nitrogen in nitro compounds to NH_3. Reid[49], using the Hengar procedure, determined Kjeldahl nitrogen on a micro-scale using an all-glass apparatus. The method gave results comparable to the macro-procedure on raw sewage and slimes. Rosicky[50] prevented foaming in the Kjeldahl procedure by addition of a mineral or fuel oil to the

flask before making it alkaline with NaOH. Zinneke[60] used a mixture of Se powder, $CuSO_4$, HgO and anhydrous Na_2SO_4 for the decomposition of nitrogen containing compounds. A special apparatus is described.

Nitrate Nitrogen

Hahn[22] found that nitrates could be determined in the presence of large amounts of calcium and magnesium if Versene was added to the KOH alkalizing solution to sequester these two elements. Broach and Testerman[6] state that in order for the reaction between nitrate and phenoldisulfonic acid to take place, the sample must be absolutely dry, the presence of hydrated or hydroxy compounds interfere to give low results due to the evolution of nitrogen oxides. De Salas[15] determined nitrate polarographically in the presence of uranyl ions (sensitivity of method not given). Nitrites must be eliminated with sulfamic acid prior to the determination. Chloride ion will not interfere but fluoride in concentrations greater than 2.5 p.p.m. caused interference. Costa[12] determined nitrates by the addition of resorcinol to an acidified sample. The mixture was heated at 112° C. in a bath of 50 percent $CaCl_2$ for 30 minutes. After standing 24-48 hours, the color is compared with standards prepared in a similar manner. The limit of detection is stated to be 0.03 gamma of nitrate nitrogen per ml. Nitrites interfere with the method. For the detection of nitrates and nitrites, Barnes[3] used as reagent for the detection of nitrates, a sulfuric acid solution of N-(1-naphthyl)ethylenediamine·HCl. A sensitivity of 2.5 gamma of nitrate N per ml. is claimed. About 0.25 gamma can be detected.

Ammonia Nitrogen

Kruse and Mellon[33] proposed the use of the pyridine-pyrazolone reagent for the determination of free NH_3. The purple color formed is extractable and the method is sensitive to 0.025 to 2.5 p.p.m. Interference by CN^-, CNO^-, and CNS^- may be removed by first passing the sample through an anion exchange resin. Sijderius[55] found that for NH_3 concentrations below 0.2 p.p.m., the relation between concentration and the extinction coefficient was not linear. When a known amount of NH_3 was added to the sample so that the reading was greater than 0.100, a straight line could be obtained after deduction of the blank. A series of standards may be made up containing increasing amounts of NH_3. The Nessler reagent of Urbach was used. Meleshko and Mazo[45] prepared ammonia-free water by passing distilled water over a sulfonated cation resin (name not given). Oxidizing substances are also removed.

Organic Compounds

Many papers were published during the year on the determination of organic compounds that might be of interest to sanitary chemists. Some of the more pertinent papers will be considered. Braus, et al[5] presented a schematic analyses for the determination of the various organic groups of compounds which might be present in industrial wastes. After adsorption of the organics on activated carbon, the carbon is dried and extracted with chloroform or ether. This extract

is separated into phenolic, acidic, amphoteric, and water soluble non-volatile compounds. The application of this scheme to the analysis of five organic industrial wastes was illustrated. Kroner et al[32] determined pyridine and pyridine-base compounds in surface waters and industrial wastes. The sample is first distilled from an alkaline medium. The pH of the distillate is adjusted to 6.8-8.0 and benzidine hydrochloride is added, followed by n-butyl alcohol. The color reaction is carried out in the alcohol layer upon the addition of cyanogen-bromide. The reaction is sensitive to 0.005 p.p.m. of pyridine and can be used in the range of 0.005 to 1.0 p.p.m. Soloway and Wilen[57] improved the ferric chloride test for phenols by using pyridine as an alkalizing reagent and extracted the color formed with either chloroform, benzene or toluene. Hill and Herndon[26] reinvestigated the use of the p-nitrosodimethylaniline method for the determination of phenols. They obtained greater sensitivity by extracting the color with either CCl_4 or isoamyl alcohol. A sensitivity for the method of 5 p.p.b. is claimed. In the determination of chlorinated o-cresols, Gardner[20] concluded that unchlorinated o-cresol and 4-chlorocresol produce compounds with similar absorption characteristics when reacted with Gibbs reagent. 6-Chloro-o-cresol and 4,6-dichloro-o-cresol also give reaction compounds with similar absorption characteristics. o-Chloro substitution products can be detected in o-cresol if phenol is absent by taking two readings with different filters. Medin and Herndon[43] determined naphthalene by extraction with chloroform and reaction with aluminum chloride. Moisture must be absent. The accuracy of the

determination is claimed to be ± 5.6 percent. Hermanowicz and Matejak[29] determined fats in sewage by evaporating the sample (acidified to pH 1.0 with HCl) to dryness. The residue is extracted with petroleum ether in a specially designed apparatus, the petroleum ether evaporated and the fat weighed. Loewus[37] improved the "anthrone" method for the determination of carbohydrates by substituting a 2 percent solution of anthrone in ethyl acetate for the formerly used reagent of 0.1-0.2 percent solution in sulfuric acid.

Color and Turbidity

The determination of color and turbidity in industrial waste waters is still in need of study. Rudolfs and Hanlon[52] made a study of the determination of color in industrial wastes in the presence of suspended solids. Their research was directed along three main lines: (a) expressing the effect of suspended solids on the color characteristics of industrial wastes, (b) using the light absorption properties of such wastes for calculating their concentration in stream water, and (c) an investigation for projecting industrial waste color characteristics to stream conditions. They present a method for expressing the color characteristics of industrial waste suspended solids in terms of the visual hue and the degree of light absorption. Beer's law was adapted to the light absorption properties of polluted streams for the purpose of calculating the concentration of two or more polluting wastes. Staats[58] reviewed the measurement

of color and turbidity, and elaborated on those methods which offer greatest promise and discussed standards for measurement of these two parameters.

Metallic Ions

The determination of metal ions in drinking waters and industrial effluents is an important field of investigation. Sacchetta and Machado[53] were able to detect 0.2 p.p.m. of arsenic by evaporating the sample to dryness and redissolving in dilute HCl. When a copper wire is inserted in the solution, a grey coloration due to formation of As_2Cu_3 is apparent.

Hansen[23] determined chromium by evaporating the sample in a Ni dish in the presence of Na_2CO_3 and KNO_3. On fusion, the Cr^{+3} is converted to Cr^{+6}. The fused mass is leached with distilled water and filtered. The filtrate is added to a 1 percent solution of $KClO_4$ containing H_2SO_4. After boiling, the color is developed by the addition of diphenylcarbazide. Saltzman[54] carried out the oxidation of Cr^{+3} in an acid solution with $KMnO_4$. The excess permanganate was destroyed by the addition of sodium azide. The color developed with diphenylcarbazide was stabilized with a phosphate buffer.

Noll and Betz[47] removed interference from iron, zinc and lead in the determination of copper by reducing the strength of the sodium diethyldithiocarbamate reagent to 1 percent of that usually used.

They also used 0.25 N NH_4OH as an alkalizing reagent. Hogl and Sulser[25] prevented the adsorption of Cu, Pb and Zn on sample containers by the addition of 0.5 percent of glacial acetic acid. This step may possibly eliminate low values previously obtained for these elements.

In the determination of calcium with the flame photometer, Kohnlein and Lucke[29] found that the presence of phosphates interfered in the method, and that they must be eliminated or the calcium precipitated as the oxalate before its determination by this method. Mackereth[38] was able to determine from 10 to 100 gamma of calcium by reacting it with an aqueous solution of picrolonic acid. The precipitate is dissolved in 60 percent ethyl alcohol and compared with standards similarly prepared. Koroleff[30] determined calcium in sea water with chloranilic acid. A 0.1 percent solution of sodium chlorinilate buffered at a pH of 7.8 is added to the sample. After standing for 2 hours the solution is filtered and the absorption read spectrophotometrically at 550 millimicrons. Waters containing more than 3 percent chloride must be diluted. Interference due to Mg is eliminated by buffering at a pH of 7.8.

Glemser and Dautzenberg[21] studied the determination of magnesium with Titan yellow and found that the amount of the reagent must be varied according to the Mg concentration. They also used a protective colloid "Polyvoil" to stabilize the colored adsorption colloid.

Hardness

In the determination of hardness by the Schwarzenbach method, one of the difficulties encountered has been the stability of the indicator solution. Distant[16] made a study of numerous solvents for Erichrome Black T, and found that the stability of this indicator was increased greatly by using either di- or triethanolamine. With either solvent, the indicator was stable for at least seven months. Brooke and Holbrook[8] found that interference with the Versenate hardness method by polyphosphates could be eliminated by first passing the water sample through a column of Amberlite IR-1B. This effectively removes both pyro- and polyphosphates. When a highly alkaline water is encountered, Azar and Browning[2] found that by neutralizing to a pH of 7.0 before adding the buffer, a satisfactory end-point could be obtained in the hardness determination. They also found that if a high bicarbonate hardness was present, that the titration should be made immediately.

Halogens

The use of chlorine is no longer confined to the field of water treatment, but is finding wide use in the industrial field. Marks and Chamberlin[40] made a study of the determination of chlorine in metal finishing wastes. They found that in the determination of Cl_2 in cyanide wastes, when the heavy metal cyanides were absent, either the amperometric titration, o-tolidine method or iodometric titration

were suitable. If heavy metal cyanides are present, then the amperometric titration is indicated. This method should also be used when chromates are present. Marks[39] also discussed the principles and application of the amperometric determination of chlorine in water and points out its advantages over other methods. The method also will determine total available bromine, but is not precise in differentiating free from combined bromine. Asmus and Garshagen[1] also studied the determination of Cl_2 or Br_2 by the well-known reaction of these halogens in the presence of pyridine and a primery amine. McEwen and Grant[42] used o-dianisidine dihydrochloride as an indicator in the determination of free chlorine.

The determination of iodine in microquantities in water solution was studied by Kramer, et al[31]. They found the amperometric method to be accurate to ± 0.01 p.p.m. in the range of iodine concentrations from 0.2 to 2.0 p.p.m. The determination can be made at the pH range usually encountered in water supplies, which is advantageous when determining germicidal properties. No evidence of the reaction between iodine and ammonia to form iodoamines was found under the experimental conditions used. Phosphate buffers interfere in the stabilization of iodine residuals.

With the increased use of fluoridization of water supplies, studies have been made on the determination of fluorides. Megregian and Maier[44] made a study of the present "standard" method and found that by modifying the zirconium-alizarin reagent, a greater sensitivity

could be obtained. Although principle emphasis is placed on the photometric reading, visual comparison could be made by modification of the reagent. Rubin[51] also proposed a modification of the "standard" method in which the readings are made at the end of 5 minutes, instead of 1 hour. He also prepared permanent color standards. Chlorine must be absent and the color reaction must take place below 45° C. Price and Walker[48] used the bleaching effect of fluorides on an aluminum-hematoxylin lake to determine up to 1.5 p.p.m. F. The pH must be closely controlled at 4.6. Sulfates and phosphates interfered to a lesser extent than with other methods. The hematoxylin must have been previously oxidized with H_2O_2 before use. Bumstead and Wells[9] determined from 0 to 100 micrograms of fluoride per 100 ml. of water by modifying the zirconium-alizarin reagent. Their reagent contained 0.30 g. of zirconium oxychloride, 0.35 g. of alizarin sodium sulfonate, 40 ml. of concentrated H_2SO_4 and 120 ml. of concentrated HCl per liter. The color was developed for two hours and readings were made spectrophotometrically at 525 millimicrons. For higher fluoride concentrations, the amounts of zirconium and alizarin in the reagent were doubled.

Cyanogen Compounds

The analytical determination of cyanide in polluted waters and industrial wastes is still under study as many variables have not been satisfactorily solved. Huditz and Flascher[27] collected considerable data to show that CN^- could be titrated with a standard

nickel solution in the presence of halides, using "murexide" as an indicator. Best results were obtained by adding an excess of nickel solution and back-titrating with a standard solution of ethylenediaminetetraacetic acid. Kruse and Mellon[34] proposed methods for the determination of cyanide, cyanate and thiocyanate in sewage. Cyanide was determined by the pyridine-pyrazolone method in the range of 0.04 to 2.0 p.p.m. The thiocyanate was complexed as a di-pyridine-copper (II) thiocyanate, cyanide interference being removed previously by boiling the acidified sample. They determined cyanate with pyridine-pyrazolone reagent at pH 3.7 (the pH used for the CN^- determination with the reagent was controlled at 6-8). Other interfering elements are removed by cation exchange resins. Brooke[7] proposed a method for the determination of CN^- which does not require preliminary distillation and is sensitive in the range of 0.5 to 3.0 p.p.m. The method is based upon the fact that when an alkaline solution of the palladium complex of alpha-furildioxime reacts with cyanide, the alpha-furildioxime is released from the complex and will react with nickel to give the familiar red color.

Sulfur Compounds

Bethge[4] developed a method for the determination of sulfide which is claimed to be as satisfactory as the well-known methylene blue procedure. The sulfide is absorbed in a zinc acetate solution. Ammonium molybdate and phosphoric acid are added, the color formed is read at 700 millimicrons. Dunicz and Rosenqvist[17] oxidized the

sulfide to sulfate with an excess of a standard solution of KClO. The excess hypochlorite was then determined iodometrically. The method can be used for determining sulfide concentrations in the range of 0.6 to 3.2 mg.

Detergents

With increased use of detergents, methods for their determination are necessary in many cases. Lewis and Herndon[36] made a survey of existing methods and chose two for investigation: (a) turbidimetric titration; and (b) two-phase dye-transfer titration. The first method for "surfaces" in dilute solution depends upon the fact that the mixing of solutions of oppositely charged colloids results in a turbidity which reaches a maximum at or near the equivalence points. Many factors tended to make this method unsatisfactory. The second method depends upon the difference in solubility of a dyestuff-surface active agent complex in water and organic solvents. This method proved to be satisfactory for the determination of "surfaces" in water and sewage.

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